

CALIBRATING THE HIGH PURITY GERMANIUM SYSTEM USED ON THE MONITORED STACKS AT TA-53

Purpose This Air Quality Group procedure describes the calibration of the High Purity Germanium detector system used to determine the isotopic compositions of the radioactive gasses being discharged from the stacks at the Los Alamos Neutron Science Center (LANSCE).

Scope This procedure applies to the calibration of the High Purity Germanium detector systems on stacks ES-2 and ES-3 at TA53. These stacks monitoring systems are part of the LANSCE radioactive air emissions monitoring project.

**In this
procedure**

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**Hazard
Control Plan**

The hazard evaluation associated with this work is documented in Attachment 1: Initial risk = **low**. Residual risk = **low**. Work permits required: none. First authorization review date is one year from group leader signature below; subsequent authorizations are on file in group office.

Signatures
(continued on
next page)

Prepared by: Robert Keys, ESH-17	Date: <u>5/7/01</u>
Approved by: Dave Fuehne, Rad-NESHAP Project Leader	Date: <u>5/21/01</u>
Approved by: Terry Morgan, QA Officer	Date: <u>5/22/01</u>
Work authorized by: Doug Stavert, ESH-17 Group Leader	Date: <u>5/23/01</u>

05/31/01

CONTROLLED DOCUMENT

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General information about this procedure

Attachments

This procedure has the following attachments:

Number	Attachment Title	No. of pages
1	Hazard Control Plan	2
2	Point source holder	1
3	Sample can holder	1

History of revision

This table lists the revision history and effective dates of this procedure.

Revision	Date	Description Of Changes
0	6/2/93	New document, issued as HS-1/TA-53-STACK-DP-003.
1	7/21/94	Updated to reflect current operations, issued as ESH-1/TA-53-STACK-DP-003.
2	7/11/96	Reformatted and updated for ES-2 modifications. Issued as ESH-17-603, R2.
3	5/20/96	Reformatted and updated for FM requirements. Issued as 53 FMP 104-03.3.
4	5/25/01	Reformatted and updated for ESH-17 modifications. Issued as ESH-17-603, R4.

Who requires training to this procedure?

The following personnel require training before implementing this procedure:

- individual assigned to perform all or part of this procedure

Training method

The training method for this procedure is **on-the-job** training by a previously trained individual and is documented in accordance with the procedure for training (ESH-17-024).

Annual retraining is required and will be by self-study (“reading”) training of this document.

General information, continued

References

The following documents are referenced in this procedure:

- ESH-17-024, “Personnel Training and Orientation”
 - MP-7-OP-9-2.01, “Building, Testing, and Filling LAMPF Gamma Cans”
 - USNRC Regulatory Guide 4.15, Revision 1, February 19, 1979.
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Note

Actions specified within this procedure, unless preceded with “should” or “may,” are to be considered mandatory guidance (i.e., “shall”).

Overview of calibration process

Overview of measurements

This document describes two measurements:

- The point source measurements at the sample volume center use the mixed gamma point source to test the detector's energy resolution and efficiency at the volume source's effective center.
 - The distributed source calibration is made with a distributed ^{85}Kr source inside a calibration can that is of the same geometry as the measurement cans. The measurement determines the volumetric source calibration as a function of distance from the detector and is used to normalize the relative efficiency curve to an absolute efficiency by the ratio of the point source measurements to that of the volume source.
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Frequency of calibration measurements

The two calibration measurements (point source calibration and volumetric source calibration) shall be performed at least twice annually. Ideally, these tests will be done once no more than 30 days prior to beam production in the LINAC and once no later than 30 days after the LANSCE operation cycle. The vertical profile measurement shall be performed after every physical configuration change of the germanium detector system.

Calibration standard for detector performance check

The point sources used to calibrate the detectors need to be obtained from a reputable company and be free of any contamination. Swipes of these sources should be taken prior to their use. If any activity is found on the swipes, the sources should be disposed of as rad waste. If other full energy peaks not associated with the isotope of interest are found, and can not be attributed to background interference, the source should be discarded and replaced with another.

Overview of calibration process, continued

Calibration standard for point source measurements

The point source calibration test requires a mixed gamma-ray certified reference source that is traceable to NIST. Certification of the source is based upon participation in the USCEA/NIST Measurements Assurance Program, which meets the quality assurance requirements of the USNRC Regulatory Guide 4.15, Revision 1, February 19, 1979.

Store all documentation for the source in the Stack Gas Studies notebook along with the other calibration documentation. Replace the source if it has less than 5 full energy photopeaks in the range of 0-3MeV of sufficient strength to yield at least 10,000 counts in 2 hours or less. The source must contain a radionuclide yielding a full energy photopeak at 514 keV.

Calibration standard for distributed source calibration

The distributed source calibration test uses a gaseous ^{85}Kr source in a volume identical to the one used in the stack measurements. The source must have been fabricated using a traceable concentration. File a copy of the documentation on how the distributed source was made and its validity for use in the Stack Gas Studies notebook along with the other calibration information including the source identification.

Environmental factors for equipment

The HPGE detector must be kept at liquid nitrogen temperatures at all times. Follow ESH-17-617 to keep dewars filled with liquid nitrogen.

Point source measurements

Background This measurement is used to determine the detector energy calibration and energy-dependent efficiency. The point source measurements are used to determine the detection system's energy response at the effective center of the volume source.

To perform these measurements, a NIST traceable mixed source is used. A source holder is also required for positioning the source at known distances and maintaining the source centered axially on the detector (shown in Attachment 2).

Certificate files In order to calibrate a system, a standard of known energies and activities is required. Use the NIST-traceable mixed-radionuclide calibration source. These standard sources come with data sheets describing the nuclides, energy lines, and activities contained within the source. A Certificate File is an electronic copy of the source certificate. The Certificate File Editor (CFE) is a program used to create and edit certificate files. Once generated, these files are used to provide a quick means for calibrating the system.

Steps to build a certificate file To build a certificate file from scratch, see the documentation on Using the Certificate File Editor in the GENIE-2000 documentation. To build a new certificate file from an existing file, perform the following steps:

Step	Action
1	Double-click on the icon for the Certificate File Editor (CFE). CFE and the Nuclide Library Editor (NLE) give you the ability to extract nuclide and energy line information from a previously created file.
2	Select <u>O</u> ptions from CFE's menu bar and select <u>C</u> ertificate Extract.
3	Click on <u>O</u> pen and select the most recent point source certificate file to use it as a template from the Files list box
4	The Extract Certificate Nuclides list box will be populated with the energy lines from the selected certificate. Click on Select all to extract all lines in the calibration source certificate file.
5	Edit the extracted Certificate File.
6	Scan the summary list box and select the lowest energy line (88.03 keV).
7	Click on the Emission Rate box and enter the appropriate emission rate in gammas per second for Cd-109, taken from the vendor's source certificate.

Steps continued on next page.

Point source measurements, continued

Step	Action
8	Move to the Rate Uncertainty % field and enter the overall uncertainty as shown on the vendor certificate as a percentage.
9	Verify that the half-life values are correct for each radionuclide in the source.
10	Click on the <u>change</u> button to effect the edits on each line.
11	At the top of the edit window, enter an assay date, quantity, and title.
12	The quantity for the source is <u>1</u> . Enter a title in the appropriate space. Save this certificate with a recognizable and unique filename using the File, Save As, function, and close the certificate file editor.

Energy calibration

Background

Energy calibration sets the relationship between the MCA channels and the photo energies in the radiation spectrum being analyzed. In Canberra parlance, the ‘detector’ defines the hardware and software parameters of the system used to collect and analyze data. These parameters are set and managed by the software interface of GENIE-2000. Once the detector is calibrated, subsequent spectra will be saved with that set of parameters copied to each new data file. The detector may have multiple efficiency calibrations reflecting different counting geometries, but each file will use the same energy/shape calibration.

Steps to collect and perform the energy calibration

To collect and perform the energy calibration, follow these steps:

Step	Action
1	Open GENIE-2000 Acquisition and Analysis program
2	Open the datasource for detector “ESx PHA”
3	The point calibration source must be counted until the peak error is less than 2%. Select the <u>Acquire On</u> Button to start collecting data.
4	After the appropriate counting statistics are achieved stop acquisition, save the spectrum in the PHA window, and again as a data file with the name in the form: YYMMDDxx.PHA (e.g., 94070601.PHA for the first PHA file created on July 6, 1994) after editing the file information page with the appropriate data.
5	Close the ESx PHA datasource, then open the newly created CAM file YYMMDDxx.PHA .
6	Select <u>Calibrate</u> , then Energy Full from menu bar, and select ‘by Certificate File’.
7	Open the current Certificate File for the point calibration source.
8	In the Energy text box, highlight the lowest energy peak listed in the table (it should be 88 keV).
9	The 88 keV peak information appears in the list box with all zeros for the associated peak parameter values. Move the cursor until it is over the 88 keV peak in the spectral window.
10	Move the cursor to the exact peak channel by using the Expand Mode (F8).
11	Once the cursor is positioned on the peak channel, click on the <u>Cursor</u> button in the Energy Calibration-Full screen.

Steps continued on next page.

Energy calibration, continued

Step	Action
12	Values for the Channel FWHM and Low Tail for 88 keV should be calculated and the highlight bar moved down to the next line of the list box. Move the highlight bar down the list to the 1836 peak in the table.
13	Return to the spectrum window and place the cursor on the 1836 peak channel (using Expand Mode), click the <u>C</u> ursor button in the calibration window.
14	Click the Accept button to accept these two peak lines.
15	The program will calculate a set of calibration curves based on these two points and present the Show Energy Calibration Curves result screen. The energy equation should show close to a 0.5 keV/ch slope.
16	More energy calibration points need to be added. Click the <u>A</u> uto button and the remainder of the peaks will be added to the calibration file.
17	Click the Show button. Print the calibration curve and select OK to save the calibration.
18	Each of the energy lines will have the associated peak parameters calculated. Click <u>S</u> how again to view the curves and coefficients, then click on OK to Energy Calibration-Full.
19	Save the energy calibration by selecting <u>C</u> alibrate, <u>S</u> ore from the menu bar and type in the Calibration file name, description, and geometry. Click OK to save and store the new calibration coefficients as a calibration file.

Efficiency calibration

Background The efficiency calibration will be performed at the effective center of the volume source. The efficiency calibration is used to perform quantitative spectral analyses.

**Steps to
obtain the
efficiency
calibration**

To obtain the efficiency calibration, perform the following steps

Step	Action
1	Ensure that the spectrum acquired when performing the energy calibration is still in the spectral display window. If it is not, open the file.
2	Select <u>C</u> alibrate then <u>E</u> fficiency, then select From Certificate File and choose the appropriate file from the file list.
4	Click on <u>A</u> uto. GENIE-2000 will then perform a peak locate with peak area analysis for each peak in the current datasource. The list box is then populated with the peak areas in the datasource that match those in the certificate file.
5	Examine the table of energies. If necessary, highlight and delete energy values of peaks with insufficient count data to calculate the efficiency at that energy. Usually, these are peaks resulting from radionuclides that have decayed away over time.
6	Click on <u>S</u> how to calculate the coefficients of the efficiency curve and view the results.
7	Select Dual and change the Order of the polynomial to 4.
8	If the curve is satisfactory, Click on the Print button to print a copy of the efficiency curve for the record.
9	Click on OK to accept the calibration.
10	Select <u>C</u> alibrate, <u>S</u> ore from the menu bar and type in the Calibration file name, description, and geometry. Click OK to save and store the new calibration coefficients as a calibration file.
11	Select Analysis Sequence, Efficiency Cal Report, and print the resulting output to get a detailed list of the energy-efficiency coefficients for the efficiency curve.

Distributed source calibration

Description

This test establishes the absolute efficiency of the system for a volume-distributed source of ^{85}Kr 514 keV gamma rays. In an identical counting geometry, this efficiency is used to count the 511 keV annihilation radiation from the stack gas. This test also provides the extra data required to convert the relative efficiency curve of the point source measurements to an absolute efficiency vs. energy curve for a distributed source. The process relies on “calibration containers” that are of the same geometry as the measurement “container” used during beam operations. The “calibration container” contains a known quantity of ^{85}Kr gas (fabricated and tested using procedure, MP-7-OP-9-2.01 “Building, Testing, and Filling LAMPF Gamma Cans.”) This procedure requires a positioning stand for centering the source “containers” over the detector in a reproducible geometry.

Check the cans for vacuum

Prior to starting the measurements of the calibration cans, place a straight edge next to the flat sides of each can. These flat sides should bow inward slightly, showing that the cans are still under vacuum and have not lost any ^{85}Kr gas. If the ends of the cans are flat, the quantity of ^{85}Kr inside is unknown and must not be used. It should be refilled using procedure MP-7-OP-9-2.01, “Building, Testing, and Filling LAMPF Gamma Cans.”

Steps to calibrate the counter

To calibrate the counter, perform the following steps:

NOTE: No adjustments should be made to the electronics from the previous measurements.

Step	Action
1	Place a dab of sealing putty on the calibration can face. When removed, its thickness will be a measure of the can-to-detector distance.
2	Place the Kr-85 calibration can on the can tripod holder and then within the detector shielding (proper geometry).
3	Open the PHA
4	Click the Acquire On button to start counting the calibration can.
5	Once the sample has been collected, edit the file information screen as previously described, and save the file as zzz.PHA (e.g., 94070602.PHA). Close the datasource.
6	Re-open the datasource just saved as a datafile.
7	Select the reporting sequence under Analyze menu which will analyze the spectrum using the DQM analysis routine, and print the results..

Steps continued on next page.

Distributed source calibration, continued

Step	Action
8	Determine the <i>detector actual efficiency</i> at 514 keV by dividing the Peak Area Counts for the 514 keV peak by the count time (to get c/s) and divide again by the calibration can activity (gammas/sec), corrected for decay ($T_{1/2} = 10.72$ y). The 514 peak area counts are located on the can activity result sheet.
9	Obtain the efficiency calculated by the software (hereafter called the <i>calculated efficiency</i>) of the 514 keV peak from the Peak Efficiency Report included in the DQM analysis printout.
10	To determine the normalization factor, divide the actual efficiency by the calculated efficiency (The resulting value should be a number > 1).
11	Correct the efficiency calibration values stored in the software to account for the volume source by selecting Calibrate, by Calibration File from the menu bar, then load the point source calibration file just created and saved.
12	Multiply each of the measured efficiency terms as a function of energy by the normalization factor, edit the efficiency values listed in the table to reflect the volume-corrected efficiency, and click to accept all changes when finished.
13	Save this file using <u>Calibrate</u> , then <u>Store</u> the file using the date of calibration as the filename (e.g., 940706.CAL) and entering an appropriate description of the file parameters. Click OK to accept and save.
14	Analyze the Kr-85 source spectrum as a DQM acquisition, verify the reported activity and reported peak efficiency values are correct, and print the results.
15	Save the calibration in the PHA datasource by opening the PHA datasource, load the new calibration file, then save the PHA datasource. Remember to always record the actual procedures performed along with the file names in the Stack logbook.
16	Record the thickness of the dried dab of sealing putty with the data in the Stack Log Book.
17	Store all printed results in the Stack Gas Studies Notebook.

Records resulting from this procedure

Records

The following records generated as a result of this procedure are to be filed chronologically as records in the Stack Gas Studies notebook (kept in the office of the Staff Member assigned to the stacks) for the current year:

- all data collected in the calibration measurements
- all calibration certificates, raw data, notes, calculations, graphs, and final results

HAZARD CONTROL PLAN

1. The work to be performed is described in this procedure.

“Calibrating The High Purity Germanium System Used On The Monitored Stacks at TA-53”

2. Describe potential hazards associated with the work (use continuation page if needed).

All hazards as described in HCP-ESH-17-TA53-XA, as supplemented and superseded by:

- 1) Radioactive sources – handling radioactive sources used in calibration; rupture or release of source could cause contamination
- 2) Radiation exposure – exposure to low-level radiation fields from stack air & sources; accident scenarios at ES-2 stack (1L Target rupture, collect rad material in HEPA filters)

3. For each hazard, list the likelihood and severity, and the resulting initial risk level (before any work controls are applied, as determined according to LIR300-00-01.0, section 7.2)

- 1) Rad Sources: occasional / moderate = low
- 2) Rad Exposure: (accidents) remote / critical = minimal
(low level) probable / negligible = minimal

Overall *initial* risk: ☐ Minimal ☒ Low ☐ Medium ☐ High

4. Applicable Laboratory, facility, or activity operational requirements directly related to the work:

☐ None ☒ List: Work Permits required? ☒ No ☐ List:

LIR-402-706-01 “Personnel Dosimetry”

HAZARD CONTROL PLAN, continued

5. Describe how the hazards listed above will be mitigated (e.g., safety equipment, administrative controls, etc.):

1) Radioactive sources - handle rad sources with care. Mixed gamma point sources must be handled by the outer ring only. Volume (can) sources of Kr-85 gas must not be dropped or caused to "vent" the can contents. Use ALARA techniques.

2) Rad Exposure –

low level rad: wear dosimetry as directed by facility-specific training. Practice ALARA techniques when in radiation fields or handling sources.

Accident scenarios: keep access training up-to-date; be aware of alarms; wear appropriate dosimetry as required by limited-access training.

6. Knowledge, skills, abilities, and training necessary to safely perform this work (check one or both):



Group-level orientation (per ESH-17-032) and training to this procedure.



Other → See training prerequisites on procedure page 3. Any additional describe here: TA-53 Facility Specific Training to have unescorted access to site
TA-53 Limited Access Area training to have access to ES-2 stack station (Building 7, room 200)

7. Any wastes and/or residual materials? (check one) ☒ None ☐ List:

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8. Considering the administrative and engineering controls to be used, the *residual* risk level (as determined according to LIR300-00-01.0, section 7.3.3) is (check one):



Minimal



Low



Medium (requires approval by Division Director)

9. Emergency actions to take in event of control failures or abnormal operation (check one):



None



List:

During LANSCE accelerator operation, the Central Control Room (CCR) and ESH-1 offices are staffed 24 hours, 7 days. Contact these offices for assistance as needed.

CCR: 667-5729; Building 4, room 203.

ESH-1 Field Office: 667-7069, Building 395, room 101.

Signature of preparer of this HCP: This HCP was prepared by a knowledgeable individual and reviewed in accordance with requirements in LIR 300-00-01 and LIR 300-00-02.

Preparer(s) signature(s)

Name(s) (print)

/Position

Date

Signature by group leader on procedure title page signifies authorization to perform work for personnel properly trained to this procedure. This authorization will be renewed annually and documented in ESH-17 records.

Controlled copies are considered authorized. Work will be performed to controlled copies only. This plan and procedure will be revised according to ESH-17-022 and distributed according to ESH-17-030.

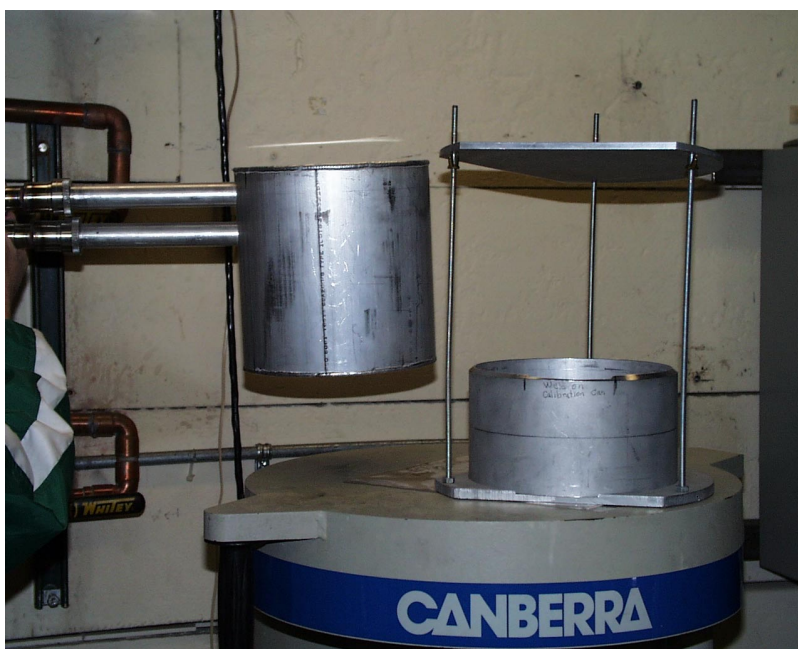
POINT SOURCE HOLDER



SAMPLE CAN HOLDER



Stack Gas Sample Can locked in the sample can holder.



Stack Gas Sample Can and empty sample can holder. The lid of the holder must be removed to insert the sample can onto the support ring, then the lid inserted onto the locking rods and tightened with the three wing nuts.